

JUL 07 2008

PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: MICHEL, et al. Docket: 2002DE142  
Serial No. 10/533,999 Group Art Unit: 1796  
Filed: 05/04/2005 Examiner: Khan, Amina S  
For: Blue Dye With Particularly High Purity And Positive  
Triboelectric Control Effect

DECLARATION UNDER 37 CFR 1.132

I, Dr. Hans-Tobias Macholdt, state that I am a resident of D-64297 Darmstadt-Eberstadt, Federal Republic of Germany; that I am a citizen of the Federal Republic of Germany; that I am a chemist having graduated at the University of Darmstadt, Federal Republic of Germany; that I am one of the inventors of U.S. Patent Application Serial No. 10/533,999; for "Blue dye with particularly high purity and positive triboelectric control effect"; that I am one of the inventors of US 5,061,585 and of US 6,168,895 cited as prior art against before mentioned US Patent Application; that I consider myself qualified , by my knowledge of chemistry, and especially of triphenylmethane dyes, electrophotographic toners and developers and by my 20 years' experience in this field; that I can make the following observations and statements to wit:

**(A) The history of triphenylmethane dyes:**

The triphenylmethane dyes (or "triphenylosanilines") are an old series of blue dyes invented in 20s of the last century. The German Hoechst Aktiengesellschaft (whose successor company in this technical field Clariant is) has achieved high technical expertise in manufacturing these dyes, improving production methods and development for various industrial uses. In the year 1972, US Patent 3,652,602 (Schafer et al. Hoechst AG) issued for "Dyestuffs of the triphenylrosaniline series and process for their preparation." This patent discloses a method for producing triphenylmethane dyes with improved yields.

During the last decades triphenylmethane dyes have reached greater industrial importance in the production of blue printing inks and as colorant in blue

electrophotographic toners for xerocopy. Therefor, US Patent 5,061,585 (Macholdt et al) issued in the year 1991 for "Blue coloring agent for electrophotographic copying processes with positive control action." As environmental regulations already existed at that time, we could not use products made according to the old process of US '602 (Schafer et al), but we relied on products (see col. 4, line 11-12 in Macholdt et al.) obtained by the preparation method described in German Patent 1,919,724 (= US 3,671,553, Papenfuhs et al. Hoechst AG) issued for "Process for the preparation of very pure monosulfonic acids of triphenylmethane dyestuffs." (US '553 is also mentioned on page 1 of the specification of U.S. Patent Application Serial No. 10/533,999). This method comprises a sophisticated sequence of precipitation, filtering, washing and distillation steps and leads to products having a very high tinctorial strength (col. 2, line 72-73) due to an "excellent purity" (col. 2, line 71-72). To my knowledge, the dyes obtained according to US '553 were the purest and highest quality triphenylmethanes known before the invention of U.S. Patent Application Serial No. 10/533,999. These US '553 qualities were also used in the triphenylmethane dyes used in our US 6,168,895 (Metz, Macholdt).

**(B) The technical problem underlying U.S. Patent Application Serial No. 10/533,999:**

While environmental and health regulation became more and more stringent in the last years, it was found by HPLC analytics that US '553 dyes still contain up to 1% (= 10,000 ppm) of anilines as residue from synthesis which persist in the product despite of the numerous purification measures described. A reproduction of Example 9 of US '553 revealed an aniline content of 9,000 ppm. As anilines, especially aniline and m-toluidine, are hazardous to health at that concentration there was a need to develop products having lower aniline content than the critical limit of 2,000 ppm.

**(C) The solution of the technical problem (= the invention):**

Matching this goal was not a trivial problem since customary purification steps, such as washing, distillation, recrystallizing, combinations thereof and with US '553 process steps did not result in a sufficient reduction of aniline content. On the other

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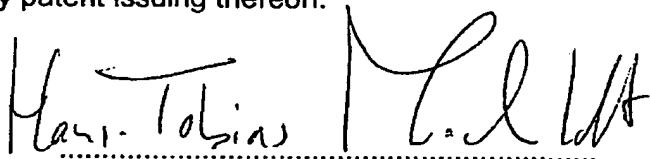
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side, we were not aware of any products on the market fulfilling these low-aniline requirements.

After laborious investigation, me and my co-inventors, surprisingly, found a new method providing the desired low-aniline products. This method is specified in claim 4 of U.S. Patent Application Serial No. 10/533,999. As this method is new and non-obvious over prior art, and, as explained above, no common method for achieving such products existed at the time this invention was made, such products were new to the world and non-obvious over the existing prior art.

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Frankfurt on the Main  
26 June 2008

A handwritten signature in black ink, appearing to read "Hans-Tobias Macholdt". The signature is fluid and cursive, with "Hans-Tobias" on the left and "Macholdt" on the right.

(Hans-Tobias Macholdt)